

# WEST Search History

DATE: Thursday, August 21, 2003

**Set Name Query**  
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*DB=USPT,PGPB,JPAB,EPAB,DWPI; THES=ASSIGNEE; PLUR=YES;  
OP=ADJ*

L13	L12 and (regenerat\$3 or activat\$3 or enhancement)	11	L13
L12	L11 and (acetic acid or citric acid or succinic acid or malonic acid or formic acid)	11	L12
L11	L10 and impregnat\$3	35	L11
L10	L9 and (ammonium nitrate or ammonium acetate or ammonium carbonate or ammonium formate)	87	L10
L9	hydrocarbon near1 synthesis	1999	L9
L8	L7 and impregnat\$3	24	L8
L7	L5 and fischer tropsch	35	L7
L6	L5 and fischer near1 tropsch	0	L6
L5	L4 and cobalt	535	L5
L4	L3 and ammonium salts	1336	L4
L3	regenerat\$3 with catalyst	20683	L3

*DB=USPT,PGPB; THES=ASSIGNEE; PLUR=YES; OP=ADJ*

L2	L1 and impregnat\$3	1	L2
L1	4795726.pn.	1	L1

END OF SEARCH HISTORY

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L13: Entry 7 of 11

File: USPT

Oct 10, 2000

DOCUMENT-IDENTIFIER: US 6130184 A

TITLE: Cobalt based fischer-tropsch catalyst

Brief Summary Text (1):

The present invention relates to a new process for the preparation of a catalyst or catalyst precursor, the catalyst or catalyst precursor thus obtained and a process for the preparation of hydrocarbons from synthesis gas using the new catalyst or catalyst precursor.

Brief Summary Text (4):

Typically, the catalysts in the prior art have been prepared by impregnation of a porous carrier with one or more soluble cobalt salts and a quantity of a solvent, followed by drying, calcination and optionally activation. In the case of pore impregnation of a porous carrier, it will usually be possible to start with a mechanical strong extrudate. However, the maximum cobalt loading that can be obtained by a single impregnation step is restricted by the pore volume of the carrier and the solubility of the cobalt salt. In practice, several impregnation steps are needed to obtain the desired quantity of cobalt. The need for such a number of steps is undesirable for the preparation of catalysts on a commercial scale.

Brief Summary Text (19):

The cobalt compound which is at least partially insoluble in the liquid may be obtained by precipitation. Any precipitation method known in the art may be used. Preferably, the cobalt compound is precipitated by addition of a base or a base-releasing compound to a solution of a soluble cobalt compound, for example by the addition of sodium hydroxide, potassium hydroxide, ammonia, urea, or ammonium carbonate. Any suitable soluble cobalt compound may be used, preferably cobalt nitrate, cobalt sulphate or cobalt acetate, more preferably cobalt nitrate. Alternatively, the cobalt compound may be precipitated by the addition of an acid or an acid-releasing compound to a cobalt ammonia complex. The precipitated cobalt compound may be separated from the solution, washed, dried, and, optionally, calcined. Suitable separation, washing, drying and calcining methods are commonly known in the art.

Brief Summary Text (29):

To obtain strong extrudates, it is preferred to include in the mixture, prior to extrusion, at least one compound which acts as a peptising agent for the titania. Suitable peptising agents for inclusion in the extrudable mixture are well known in the art and

include basic and acidic compounds. Examples of basic compounds are ammonia, ammonia-releasing compounds, ammonium compounds or organic amines. Such basic compounds are removed upon calcination and are not retained in the extrudates to impair the catalytic performance of the final product. Preferred basic compounds are organic amines or ammonium compounds. A most suitable organic amine is ethanol amine. Suitable acidic peptising agents include weak acids, for example formic acid, acetic acid, citric acid, oxalic acid, and propionic acid.

Brief Summary Text (37):

The present invention also relates to a cobalt-containing catalyst or catalyst precursor obtainable by a process as hereinbefore defined. The catalyst according to the present invention is typically used to catalyse a process for the preparation of hydrocarbons from synthesis gas. Typically, when in use in that process, at least part of the cobalt is in its metallic state.

Brief Summary Text (38):

Therefore, it is normally advantageous to activate the catalyst or catalyst precursor prior to use by a reduction treatment, in the presence of hydrogen at elevated temperature. Typically, the reduction treatment involves treating the catalyst at a temperature in the range of from 100 to 450.degree. C. for 1 to 48 hours at elevated pressure, typically from 1 to 200 bar abs. Pure hydrogen may be used in the reduction treatment, but it is usually preferred to apply a mixture of hydrogen and an inert gas, like nitrogen. The relative amount of hydrogen present in the mixture may range between 0 and 100% by volume.

Brief Summary Text (40):

If possible, it is preferred to activate the catalyst in-situ, that is inside the reactor. International patent application publication No. WO 97/17137 describes an in-situ catalyst activation process which comprises contacting the catalyst in the presence of hydrocarbon liquid with a hydrogen-containing gas at a hydrogen partial pressure of at least 15 bar abs., preferably at least 20 bar abs., more preferably at least 30 bar abs. Typically, in this process the hydrogen partial pressure is at most 200 bar abs.

Brief Summary Text (41):

It is advantageous to rejuvenate spent catalyst, i.e. catalyst that has lost at least part of the initial activity of an activated fresh catalyst, by subjecting it to a ROR treatment. Typically, the ROR treatment involves the steps, in sequence, of reduction with a hydrogen-containing gas, oxidation with an oxygen-containing gas, and reduction with a hydrogen-containing gas.

Brief Summary Text (47):

Further, it will be understood that the skilled person is capable to select the most appropriate conditions for a specific reactor configuration and reaction regime. For example, the preferred gas hourly space velocity may depend upon the type of reaction regime that is being applied. Thus, if it is desired to operate the hydrocarbon synthesis process with a fixed bed regime, preferably the gas hourly space velocity is chosen in the range from 500 to 2500 Nl/l/h. If it is desired to operate the hydrocarbon synthesis

process with a slurry phase regime, preferably the gas hourly space velocity is chosen in the range from 1500 to 7500 Nl/l/h.

Detailed Description Text (6):

A solution was prepared containing 100 g  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and 4 g  $\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and 10 ml of water. 70 g of the titania extrudates were impregnated with this solution in four impregnation steps. After each impregnation step the extrudates were dried at 120 .degree. C. for 16 hours and calcined at 500 .degree. C. for two hours. The resulting impregnated and calcined extrudates.

Detailed Description Text (16):

Commercially available titania powder (P25 ex. Degussa) was mixed with water. The mixture contained 30% by weight of titania powder. The mixture was spray-dried using a Niro Atomizer. The inlet temperature was 250.degree. C. and the outlet temperature was 117.degree. C. The resulting product was calcined for 1 hour at 500.degree. C. The spray-dried titania particles were impregnated with a concentrated solution containing cobalt nitrate and manganese nitrate. The solution was prepared by heating solid cobalt nitrate ( $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) and solid manganese nitrate ( $\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ) to a temperature of 60.degree. C., thus causing the metal nitrates to dissolve in their own crystal water. The impregnated titania particles were dried for 2 hours at 120.degree. C. and subsequently calcined in air for 1 hour at 400.degree. C. The resulting catalyst particles contained 20 wt % Co and 1 wt % Mn.